

1664

RESULTS OF EXPERIMENTS  
TO DOCUMENT  
THE PHYTOTOXICOLOGY  
OF BLACK LIQUOR

AUGUST 1991



Environment  
Environnement

201 5/91



RESULTS OF EXPERIMENTS TO DOCUMENT THE  
PHYTOTOXICITY OF BLACK LIQUOR

ARB-083-91-PHYTO

Report prepared by:  
W.D. McIlveen  
Phytotoxicology Section  
Air Resources Branch  
Ontario Ministry of the Environment

AUGUST 1991



Cette publication technique  
n'est disponible qu'en anglais.

Copyright: Queen's Printer for Ontario, 1991  
This publication may be reproduced for non-commercial purposes  
with appropriate attribution.

PIBS 1664  
log 91-2231-083



# **RESULTS OF EXPERIMENTS TO DOCUMENT THE PHYTOTOXICITY OF BLACK LIQUOR**

## **INTRODUCTION**

The production of paper products in the Kraft process involves the removal of lignin from the pulpwood thereby leaving the desirable cellulose component. This process requires digestion of the wood in a combination of sodium sulphide and sodium hydroxide, known as "white liquor". As the process proceeds, the white liquor acquires substantial quantities of lignins and other materials and is called "black liquor". The black liquor is of variable consistency and concentration at different periods of the process.

Black liquor is a waste product that presents certain difficulties in disposal. At low rates, it can be used to suppress road dust. It is, however, toxic to plants at higher rates. Over the years, the Phytotoxicology Section has investigated several incidents where damage to vegetation caused by this material has occurred. These incidents included discharges of black liquor to the atmosphere and excessive application rates to a road surface. Experimental exposures of plants to black liquor have shown that the material is phytotoxic to several plant species.

In the present case, off-site seepage of black liquor from a holding lagoon at a container board recycling plant at Trenton has been identified as a problem. Staff from the Investigations and Enforcement Branch (IEB), Ontario Ministry of the Environment requested that the Phytotoxicology Section assist in documenting the phytotoxicity of the black liquor at this site. This report is a summary of the information derived to date in the follow-up study.

## **METHODS**

A sample of black liquor (consisting of several bottles) from the holding lagoon in question was provided by staff of IEB. This was used in subsequent tests conducted by staff of the Phytotoxicology Section. These tests included both greenhouse exposures of growing plants and of germination tests in the laboratory. These tests are described individually below.

### **Experiment 1.**

The first experiment was conducted in the controlled environment greenhouse located at Brampton. In this experiment, established 22-day old bean (*Phaseolus vulgaris* cv Dark Red Kidney) and radish (*Raphanus sativus* cv Cherry Belle) plants were exposed to black liquor treatments. Each pot contained three plants grown in greenhouse potting soil. Three treatments were applied. These included i) a soil drench consisting of 100 ml of black liquor poured onto the soil surface, ii) a light spray of pure black liquor sufficient to wet the foliage and iii) an untreated control. Each treatment was replicated 5 times.

The treated plants were maintained under natural light supplemented with fluorescent lamps for 16 hour per day at 22 C. The condition of the plants was monitored daily and injury to foliage was assessed at 11 days after treatment. Fresh weights of radish plants were determined the following day.

## **Experiment 2**

The second experimental conditions were similar to those of Experiment 1. In this instance, only bean plants were used and the treatments involved only soil drenches. The drenches included 100 ml of a graded series of black liquor diluted with distilled water. The concentrations of black liquor included 0%, 0.2%, 0.5%, 1%, 2%, 5%, 10%, 20%, 50% and 100% and each treatment was replicated 5 times. The condition of plants was recorded after 22 days. Foliar injury (necrosis and chlorosis), number of pods, fresh weights of shoots, roots and pods and dry weights of roots and shoots were determined.

## **Experiment 3**

The third experiment was conducted in the laboratory and involved germination and root growth of seedlings. Clean sifted sand (15 g) was placed in the bottom of glass Petri dishes and covered with 2 layers of filter paper. The black liquor sample was diluted with distilled water to provide concentrations of 100%, 50%, 10%, 2%, 1%, 0.5% and 0.0%. To each of two replicate dishes for each species and concentration, 10 ml of test solution were added. After 2 hours, 20 seeds of radish and lettuce (*Lactuca sativa* cv Grand Rapids) were placed onto the filter paper in the Petri dishes. The dishes were maintained in an incubator set at 20 °C in the dark. After 9 days, the germination rates and length of seedling root were determined.

## **RESULTS**

### **Experiment 1**

The amount of injury observed on bean and radish in the first experiment is summarized in Table 1. The drench treatment killed 33% of the bean plants and 60% of the radish plants. The remaining primary foliage was severely injured on both species. Lesser necrosis was noted on the trifoliate foliage of bean. Chlorosis (yellowing) was also prominent in the plants receiving the drench treatment. While the spray treatment also resulted in moderate foliar necrosis (bean 26%, radish 13%), the extent was less severe than the drench treatment. No injury was observed in the control treatment.

Mean fresh weights of radish roots and shoots and bean shoots subjected to the spray treatment were slightly lower than for the controls. The fresh weights of shoots for bean were reduced by 64% in the drench treatment. A similar reduction of about 61% was noted for shoots of drenched radishes. An even greater reduction (85%) in radish root weight was recorded for drenched plants.

### **Experiment 2**

In the treatments which received the 100% or 50% (and to a lesser extent 20%) black liquor, necrosis similar to that found in the first experiment was observed on the bean plants (Appendix). Some necrosis of foliage was also seen in the 2% treatment; however, this injury did not appear to be related to the black liquor treatment. Although there is considerable variability in fresh and dry weights of plants, it is clear that the lowest mean weights were associated with plants receiving the highest rates of black liquor. The mean dry weight for plants in each treatment are shown in Fig. 1.

The variability in root and shoot weight is shown in Figs. 2 and 3, respectively. The upper and lower limits shown are + or - 2 standard deviations from the mean. The variability is due in part to resprouting of some plants which had exhibited initial injury.

### **Experiment 3**

The response in germination and root growth of lettuce and radish are demonstrated in Figs 4 and 5. Inhibition of radish germination occurred at about 2% black liquor while germination of lettuce was first noted at 10% black liquor. No germination occurred at 50% or over black liquor.

Root growth of both species was substantially inhibited at 10% black liquor. A noticeable reduction in growth of lettuce roots was also seen at the 2% black liquor concentration thereby indicating that lettuce roots are more sensitive to black liquor than are those of radish.

### **CONCLUSIONS**

The present experiments have demonstrated that black liquor is indeed toxic to plants, particularly to the roots. Toxicity to foliage and to germination was also demonstrated. The study did not attempt to determine the toxic component(s) in the black liquor but sodium residues from the original white liquor are likely involved. This should be resolved in any future study.

Until actual concentrations of black liquor in runoff or spill contaminated soils have been documented in the field, it is difficult to establish a comparable greenhouse study. Substantial death and injury to plants was demonstrated using soil drenches of 100 ml in 3.1 kg of soil mix (or 3.2% v/wt). In the field, seepage areas could contain up to 50% of the soil volume occupied by black liquor. The maximum treatments applied in these experiments is, therefore, quite conservative with respect to potential field conditions. On the basis of these estimates and experimental findings, it can be concluded that black liquor will cause injury to plants growing in areas that are subjected to seepage of this material.

### **REFERENCES**

Griffin, H.D. 1976. Air Quality - Fort Frances, Annual Report 1975. Ontario Ministry of the Environment, Northwestern Region.

Temple, P.J. and R.A. Richards. 1978. Effects of atmospheric deposition of sodium sulfate on bean and tomato plants. Bull. Envir. Contam. & Toxic. 19: 257-263.

Table 1. Summary of injury to foliage of bean and radish plants treated with black liquor drench or spray. Results from Experiment 1.

Species	Treatment	Necrosis			Chlorosis	
		% Plant Dead	% Primary Leaves	First Trifoliolate	Primary Leaves	First Trifoliolate
Bean	CONTROL	0.0	0.0	0.0	0.0	0.0
	SPRAY	0.0	26.7	7.7	0.0	4.7
	DRENCH	33.3	92.0	50.0	15.3	12.3

	Treatment	% Plant Dead	% Necrosis Leaves	Weight/ Plant	
				Shoot	Root
Radish	CONTROL	0.0	0.0	4.97	8.33
	SPRAY	0.0	13.3	4.41	7.03
	DRENCH	60.0	64.0	1.95	1.23

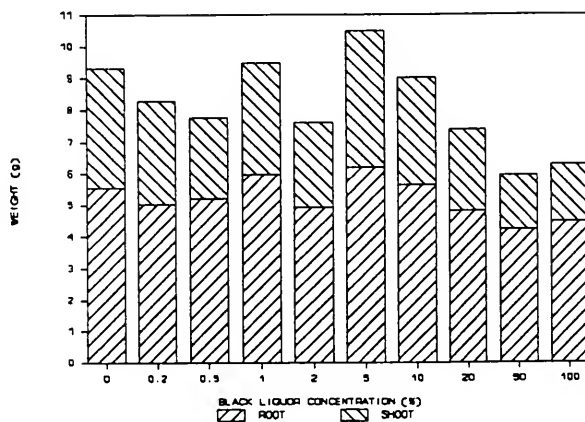
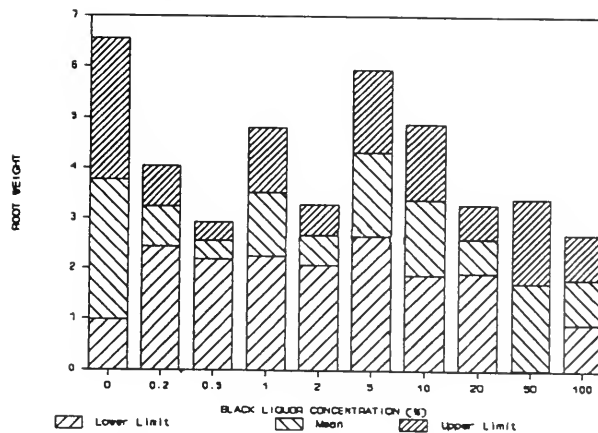
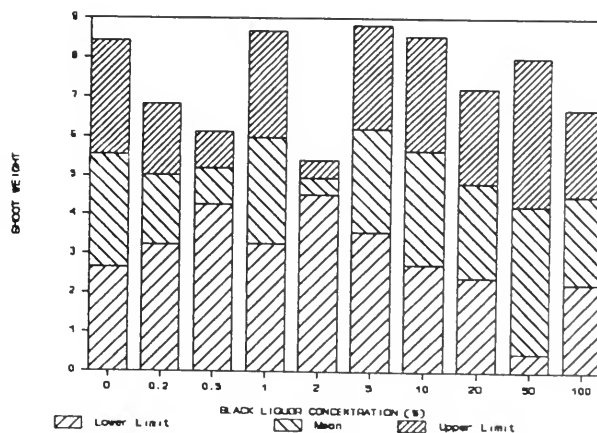


Figure 1 Weights of roots and shoots of bean plants exposed to black liquor - Experiment 2

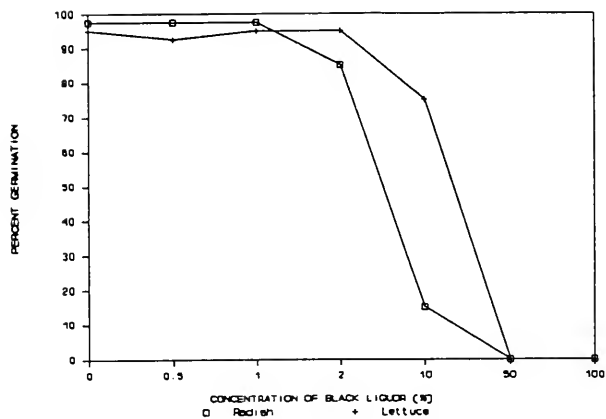




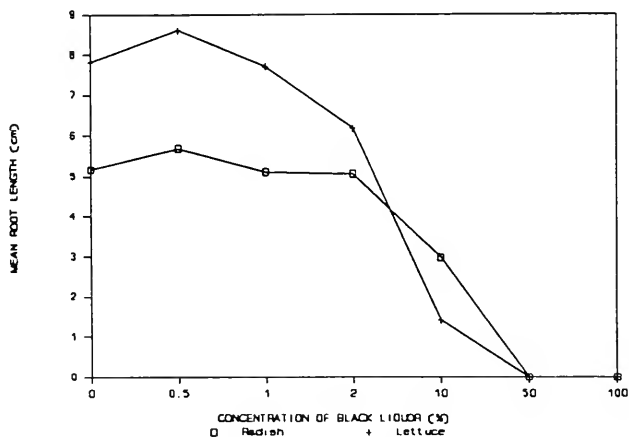
**Figure 2** Range in weights of bean roots exposed to black liquor - Experiment 2



**Figure 3** Range in weights of bean shoots exposed to black liquor - Experiment 2



**Figure 4** Effect of black liquor on the rate of germination of radish and lettuce seeds.



**Figure 5** Effect of black liquor on growth of radish and lettuce roots

# APPENDIX I.

Mean values of injury and weights of bean plants exposed to various concentrations of black liquor - Results for Experiment 2.

Conc. <sup>1</sup> (%)	Foliar Nec. <sup>2</sup>		Foliar Chlor. <sup>3</sup>		No. Pods		Fresh Shoot		Fresh Root		Fresh Pod		Dry Shoot		Dry Root		Total Wt. / Pod	
							Wt.		Wt.		Wt.		Wt.		Wt.			
0	1.2	5.2	6.4	30.6	34.3	5.4	5.542	3.771	70.3	0.779								
0.2	5.4	8.6	8.4	28.0	33.0	9.9	5.025	3.247	71.0	1.237								
0.5	0.0	5.0	8.2	27.8	27.9	7.6	5.200	2.559	63.3	0.957								
1	0.4	7.6	9.2	32.1	33.0	12.0	5.969	3.517	77.1	1.287								
2	12.2	10.6	6.4	26.3	28.3	7.6	4.944	2.664	62.2	1.214								
5	5.0	24.0	10.6	32.9	38.5	13.5	6.192	4.303	85.0	1.190								
10	3.8	5.2	7.0	29.0	32.9	8.4	5.633	3.380	70.2	1.152								
20	8.2	7.6	5.0	26.5	26.8	6.9	4.815	2.591	60.3	1.207								
50	43.0	4.4	6.4	23.7	19.4	6.3	4.240	1.711	49.3	0.877								
100	76.0	12.0	3.8	21.8	17.0	3.2	4.476	1.802	42.0	0.682								

<sup>1</sup> Concentration is ml of black liquor in 100 ml water.

<sup>2</sup> % of leaf primary leaf area with necrotic (dead) tissue

<sup>3</sup> % of leaf primary leaf area with necrotic (dead) tissue

<sup>4</sup> Total for fresh weights of roots, shoots and pods





